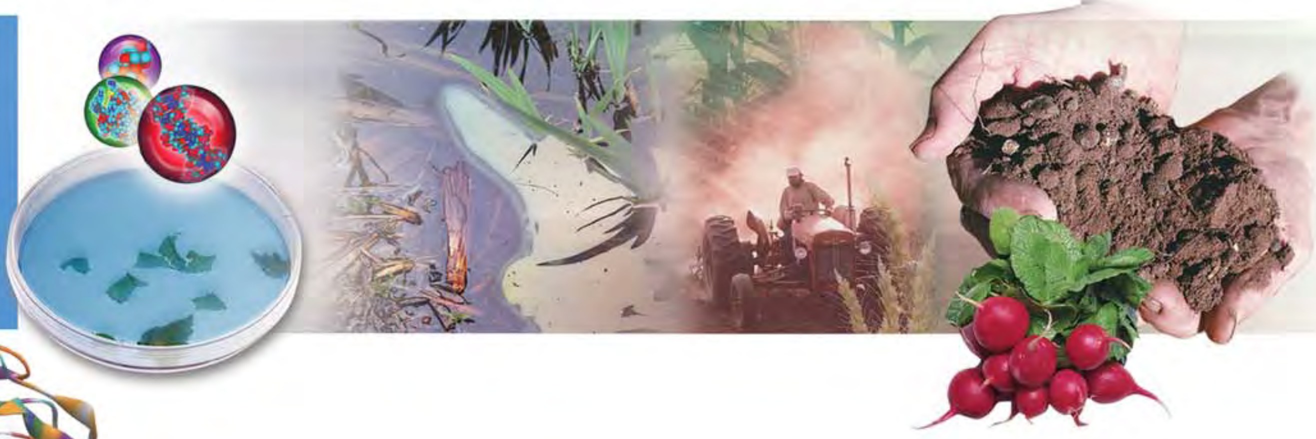
Errata

Product Manual for Dionex IonPac™ Fast Anion IIIA Columns
065152-03

For new orders of the following parts discussed in this manual, please use the updated part numbers listed below.

Part	**Old Part Number in this manual**	**Updated Part Number to use for new orders**
PROD,COL,IP,ATC-3,4X35MM	*059661*	*079932*



PRODUCT MANUAL

IonPac® Fast Anion IIIA

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PRODUCT MANUAL

for the

IONPAC® FAST ANION IIIA ANALYTICAL COLUMN
(3 x 250 mm, P/N 062964)

IONPAC® FAST ANION IIIA GUARD COLUMN
(3 x 50 mm, P/N 062966)

Document No. 065152

Revision: 03

24 November 2008

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SECTION 1 - INTRODUCTION

The IonPac® Fast Anion IIIA Analytical Column in combination with the IonPac Fast Anion IIIA Guard Column is designed for the analysis of inorganic anions including fluoride, chloride, nitrate, sulfate, phosphate, and citrate in less than 7 minutes using an isocratic hydroxide eluent delivered with an EG40, EG50, ICS-2000, or ICS-3000. Using an isocratic hydroxide eluent, the inorganic anions can easily be separated in a variety of soft drinks including carbonated beverages. The IonPac Fast Anion IIIA is compatible with pH 0-14 eluents, and eluents containing organic solvents from 0 to 100% in concentration. It can be used with any suppressible ionic eluent that does not exceed the capacity of the suppressor. Additionally, the IonPac Fast Anion IIIA has nominal efficiency of at least 6,000 plates/column for sulfate using standard operating conditions. The maximum operating pressure should be less than 3,000 psi.

Table 1
IonPac Fast Anion IIIA Packing Specifications

Column	Particle Diameter µm	Substrate X-linking %	Column Capacity µeq/column	Functional Group	Hydrophobicity
IonPac Fast Anion IIIA Analytical Column 3 x 250 mm	7.5	55	55	Alkanol quaternary ammonium	Ultra Low
IonPac Fast Anion IIIA Guard Column 3 x 50 mm	11	55	1.0	Alkanol quaternary ammonium	Ultra Low

Table 2
IonPac Fast Anion IIIA Operating Parameters

Column	Typical Back Pressure psi (MPa)	Standard Flow Rate mL/min	Maximum Flow Rate mL/min
IonPac Fast Anion IIIA 3-mm Analytical Column	≤1,800 (12.41)	1.0	2.0
IonPac Fast Anion IIIA 3-mm Guard Column	≤400 (2.76)	1.0	2.0
IonPac Fast Anion IIIA 3-mm Column Set	≤2,200 (15.17)	1.0	2.0

NOTE

1. Analytical Column resin composition: supermacroporous polyvinylbenzyl ammonium polymer cross-linked with divinylbenzene.

2. Guard Column resin composition: microporous polyvinylbenzyl ammonium polymer cross-linked with divinylbenzene.

Assistance is available for any problem that may be encountered during the shipment or operation of Dionex instrumentation and columns through the Dionex North America Technical Call Center at 1-800-DIONEX-0 (1-800-346-6390) or through any of the Dionex Offices (listed in "Dionex Worldwide Offices" on the Dionex Reference Library CD-ROM, P/N 053891).

SECTION 2 - ION CHROMATOGRAPHY SYSTEMS

The proper configuration of an Ion Chromatography System (ICS) in 2-mm or 4-mm format is based on the ratio of the 2-mm to 4-mm column cross-sectional area (a factor of 1/4). The selected format will affect the type of pump recommended. A gradient pump is designed to blend and pump isocratic, linear, or gradient mixtures of up to four mobile phase components at precisely controlled flow rates. An isocratic pump is for applications not requiring gradient and multi-eluent proportioning capabilities. Both are offered in either standard bore or microbore options.

- For an ICS in 2-mm format, Dionex recommends a microbore isocratic pump, standard bore isocratic pump, microbore gradient pump, or standard bore gradient pump .
- For an ICS in 4-mm format, Dionex recommends a standard bore isocratic pump or standard bore gradient pump.

See Appendix B, "Configuration" for specific recommended settings and parts including pumps, eluent flow rate, Self-Regenerating Suppressor (SRS), MicroMembrane Suppressor (MMS), injection loop, system void volume, detectors, and tubing back pressure.

SECTION 3 - INSTALLATION SECTION

3.1 System Requirements

3.1.1 System Requirements for 3-mm Operation

The IonPac Fast Anion IIIA 3-mm Guard and Analytical Columns are designed to run on Dionex Ion Chromatographs equipped with suppressed conductivity detection. Isocratic analyses at flow rates of 0.5 mL/min or greater can be performed on a pump with standard (1/8" pistons) pump heads. A microbore pump (1/16" pistons) must be employed for isocratic analyses at flow rates below 0.5 mL/min and gradient analyses.

3.1.2 System Void Volume

When using 3-mm columns, you should minimize system void volume. The system void volume should be scaled down to at least 1/2 of the system volume in a standard 4-mm system. For best performance, all of the tubing installed between the injection valve and detector should be PEEK tubing 0.005" ID (P/N 044221), or PEEK tubing 0.010" ID (P/N 042260). Minimize the lengths of all connecting tubing and remove all unnecessary switching valves and couplers.

3.2 The Sample Concentrator

The Trace Anion Concentrator Low Pressure Column (TAC-LP1, P/N 046026), Trace Anion Concentrator Ultra Low Pressure Column (TAC-ULP1, P/N 061400), Ultra Trace Anion Concentrator Low Pressure Column (UTAC-LP1, P/N 063079), Ultra Trace Anion Concentrator Ultra Low Pressure Column (UTAC-ULP1, P/N 063475), or Ultra Trace Anion Concentrator Extremely Low Pressure Column (UTAC-XLP1, P/N 063459) can be used for trace anion concentration work. The function of a concentrator column in these applications is to strip ions from a measured volume of a relatively clean aqueous sample matrix. This process concentrates the desired analyte species onto the concentrator column, which will lower the detection limits by 2-5 orders of magnitude. The concentrator column is used in lieu of the sample loop. Pump the sample onto the concentrator column in the opposite direction of the eluent flow. When using concentration techniques, do not overload the concentrator column by concentrating an excessive amount of sample. This can cause inaccurate results. During the concentration step, the polyvalent anions, such as phosphate and sulfate, may elute the weakly retained anions, such as fluoride and acetate, off the concentrator column.

For a detailed discussion of sample concentration techniques, refer to Section 3, “Operation,” of the Trace Anion Concentrator (TAC-LP1 and TAC-ULP1) Column Product Manual (Document No. 034972) or Section 3, “Operation,” of the Ultra Trace Anion Concentrator (UTAC-LP1, UTAC-ULP1, and UTAC-XLP1) Column Product Manual (Document No. 065091).

WARNING

IonPac Trace Anion Concentrator (TAC 2) Column (P/N 043101) is *not* optimized for use with hydroxide eluents and should *not* be used for concentrator work with the IonPac Fast Anion IIIA. In addition, the IonPac Fast Anion IIIA Guard Column has very low capacity and is not recommended as a concentrator column.

3.3 The Injection Loop

3.3.1 The 3-mm System Injection Loop, 1- 25 µL

A 1-25 µL injection loop is sufficient for most applications on a 3-mm analytical system. Generally, you should not inject more than 12.5 nanomoles of any one analyte onto a 3-mm analytical column. Injecting a larger number of moles from a sample can result in overloading the column, which can affect the detection linearity. For low concentrations of analytes larger injection loops can be used to increase sensitivity. The IonPac Fast Anion IIIA 3-mm requires a microbore IC (Ion Chromatograph) system configuration.

3.4 The IonPac Fast Anion IIIA Guard Column

An IonPac Fast Anion IIIA Guard Column is normally used with the IonPac Fast Anion IIIA Analytical Column. Retention times will increase by approximately 4% for the phosphate peak when a guard column is placed in-line prior to the analytical column. A guard is placed prior to the analytical column to prevent sample contaminants from eluting onto the analytical column. Replacing the IonPac Fast Anion IIIA Guard Column at the first sign of peak efficiency loss or decreased retention time will prolong the life of the IonPac Fast Anion IIIA Analytical Column.

3.5 Installing the CR-ATC Trap Column for Use with EGC II KOH Cartridge

For IonPac Fast Anion IIIA applications, using the Eluent Generator (EG) with EGC II KOH cartridge, a CR-ATC Continuously Regenerated Trap Column (P/N 060477) should be installed at the EGC eluent outlet. This is required for removal of trace level anionic contaminants from the carrier deionized water. See the "*Product Manual* for CR-TC (Document No. 031910)" for instructions.

As an alternative, the ATC-HC Trap Column (P/N 059604) should be installed between the pump outlet and the inlet of the EluGen Cartridge in the EG Module to remove anionic contaminants from the carrier deionized water. The ATC-HC is for use with EGC II KOH cartridge in the EG40 and EG50 Eluent Generators. See the "*Product Manual* for ATC-HC (Document No. 032697)" for instructions.

If the lower capacity ATC-3 Trap Column (P/N 059660 and 059661) is used, it should be installed between the gradient pump and the injection valve to remove anionic contaminants from the eluent. The ATC-3 column is used when performing sodium hydroxide gradient anion exchange applications using hand-prepared bottled eluents. See the "*Product Manual* for ATC-3 (Document No. 032697)" for instructions.

The ATC-HC (P/N 059604) and ATC-3 Trap Columns will require off-line regeneration. To use the ATC-HC or ATC-3 Anion Trap Columns, refer to their respective product manuals.

3.6 Eluent Storage

IonPac Fast Anion IIIA columns are designed to be used with hydroxide eluent systems. Storage under a helium atmosphere ensures contamination free operation and proper pump performance. Note: Nitrogen can be used if eluents do not contain solvents.

3.7 Anion Self-Regenerating Suppressor Requirements

An Anion Self-Regenerating Suppressor should be used for applications that require suppressed conductivity detection. It is compatible with solvent containing eluents and aqueous ionic eluents of all concentrations with which the systems and columns are compatible. Aqueous ionic eluents can be used in all ASRS ULTRA modes of operation. Solvent containing eluents should be used in the AutoSuppression External Water Mode.

IonPac Fast Anion IIIA 3-mm Analytical Columns use the ASRS ULTRA II (2 mm, P/N 061562).

For detailed information on the operation of the Anion Self-Regenerating Suppressor, see the “*Product Manual* for the Anion Self-Regenerating Suppressor ASRS ULTRA II (Document No. 031367).”

3.8 Anion Atlas Electrolytic Suppressor Requirements

An Atlas® Anion Electrolytic Suppressor (AAES) may be used instead of an ASRS ULTRA II for applications that require suppressed conductivity detection. The AAES (P/N 056116) can be used for 3 mm IonPac Fast Anion IIIA applications using eluents up to 25 µeq/min.

For detailed information on the operation of the Atlas Anion Electrolytic Suppressor, see the “*Product Manual* for the Atlas Anion Electrolytic Suppressor (Document No. 031770).”

3.9 Anion MicroMembrane Suppressor Requirements

An Anion MicroMembrane Suppressor (AMMS III) may be used instead of an ASRS ULTRA II (2-mm) for applications that require suppressed conductivity detection. Use an AMMS III 2 mm (P/N 056751) with the IonPac Fast Anion IIIA 3-mm Analytical Column. It is compatible with all solvents and concentrations with which the systems and columns are compatible.

For detailed information on the operation of the Anion MicroMembrane Suppressor, see the “*Product Manual* for the Anion MicroMembrane Suppressor III, the AMMS III (Document No. 031727).”

3.10 Using Displacement Chemical Regeneration (DCR) in Chemical Suppression Mode

Dionex recommends using the DCR Mode for chemical suppression using sulfuric acid and the AMMS III. See the “*DCR Kit Manual* (Document P/N 031664)” for details.

SAFETY: USE PROPER SAFETY PRECAUTIONS IN HANDLING ACIDS AND BASES.

3.11 Using AutoRegen with ASRS ULTRA II or AMMS III in Chemical Suppression Mode

Dionex recommends using an AutoRegen® Accessory (P/N 039594) when using the chemical suppression mode to save regenerant preparation time and reduce regenerant consumption and waste. For more detailed information on the use of the AutoRegen Accessory see the “*AutoRegen Accessory Manual* (Document No. 032853).” For more detailed information on the use of AutoRegen Regenerant Cartridges, see the “*Product Manual* for the AutoRegen Regenerant Cartridge Refills (Document No. 032852).”

3.12 Detector Requirements

See Section 2, “Comparison of Ion Chromatography Systems,” for 3-mm system detector, cell and thermal stabilizer requirements.

3.13 Using the Eluent Generator with Fast Anion IIIA

For information on the operation of the EG40 please refer to the "*Product Manual* for EG40 (Document No. 031373)." For the EG50, please refer to the "*Product Manual* for EG50 (Document No. 031908)."

SECTION 4 - OPERATION

4.1 General Operating Conditions

Sample Volume:	3-mm: 2.5 µL Loop + 0.8 µL Injection valve dead volume
Column:	3-mm: IonPac Fast Anion IIIA 3-mm Analytical + IonPac Fast Anion IIIA 3-mm Guard
Eluent:	22 mM KOH
Eluent Source:	EG40 or EG50
Eluent Flow Rate:	3-mm: 1.0 mL/min
Temperature:	30 °C
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS ULTRA II (2-mm)
or AAES Suppressor:	Anion Atlas Electrolytic Suppressor, AAES AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS III (2-mm)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	≤ 2 µS
Long-term Storage Solution (> 1 week):	100 mM Sodium Borate
Short-term Storage Solution (< 1 week):	Eluent

4.2 IonPac Fast Anion IIIA Operation Precautions

4.2.1 Filter and Degas Eluents

If you suspect particulates in your DI water supply, then it is recommended that you filter the eluents before hooking up to the system. Degassing eluents will ensure optimum performance of the pump.

4.2.2 Filter Samples

Particulates in the sample can clog the bed support. Filtering samples can avoid this problem.

4.2.3 Eluent and Sample pH

Fast Anion IIIA can handle a wide range of sample or eluent, pH (0-14). However, it is always recommended to avoid switching eluents from one extreme to another. When in doubt about the pH compatibility of the column add a DI water step when switching eluents from one extreme to another.

4.2.4 Maximum Flow Rate

The average operating IonPac Fast Anion IIIA analytical and guard column flow rate is 1 mL/min. However, the IonPac Fast Anion IIIA can handle flow rates up to 2 mL/min. Running higher than the recommended flow rate for long periods of time can cause head space to the inlet of the column.

4.2.5 Maximum Operating Pressure

The average IonPac Fast Anion IIIA analytical and guard column operating pressure is below 2200 psi. However, the column can handle up to a maximum of 3000 psi of pressure. Exposing the column above 3000 psi can cause head space to the inlet of the column.

4.3 Chemical Purity Requirements

Obtaining reliable, consistent, and accurate results requires eluents that are free of ionic impurities. Chemicals, solvents, and deionized water used to prepare eluents must be of the highest purity available. Low trace impurities and low particle levels in eluents also help to protect your ion exchange columns and system components. Dionex cannot guarantee proper column performance when the quality of the chemicals, solvents, and water used to prepare eluents has been compromised.

4.3.1 Inorganic Chemicals

Reagent Grade inorganic chemicals should always be used to prepare ionic eluents. Whenever possible, inorganic chemicals that meet or surpass the latest American Chemical Society standard for purity should be used. These inorganic chemicals will show a lot analysis on each label.

4.3.2 Deionized Water

The deionized water used to prepare eluents should be a "Type I Reagent Grade Water" with a specific resistance of 18.2 megohm-cm. The deionized water should be free of ionized impurities, organics, microorganisms, and particulate matter larger than 0.2 µm. Bottled HPLC-Grade Water, with the exception of **Burdick & Jackson**, should not be used since most bottled water contains an unacceptable level of ionic impurities.

4.3.3 Solvents

Solvents can be added to the ionic eluents used with IonPac Fast Anion IIIA columns to modify the ion exchange process or improve sample solubility. The solvents used must be free of ionic impurities; however, since most manufacturers of solvents do not test for ionic impurities, it is important that the highest grade of solvents available be used. Several manufacturers are currently making ultra high purity solvents that are compatible for HPLC and spectrophotometric applications. These ultra high purity solvents will usually ensure that your chromatography is not affected by ionic impurities. At Dionex, we have obtained consistent results using High Purity Solvents manufactured by **Burdick and Jackson** and Optima® Solvents by **Fisher Scientific**.

When using a solvent in an ionic eluent the column generated back pressures will depend on; the solvent used, concentration of the solvent, the ionic strength of the eluent, and the flow rate used. The column back pressure will vary as the composition of water-methanol and water-acetonitrile mixture varies. The practical back pressure limit for the IonPac Fast Anion IIIA columns is 3,000 psi (20.68 MPa).

The IonPac Fast Anion IIIA can withstand common HPLC solvents in a concentration range of 0–100%. Solvents and water should be premixed in concentrations which allow proper mixing by the gradient pump and minimizes outgassing. Ensure that all of the inorganic chemicals are soluble in the highest solvent concentration to be used during the analysis.

Table 3
HPLC Solvents for Use with IonPac Fast Anion IIIA Columns

Solvent	Maximum Operating Concentration
Acetonitrile	100%
Methanol	100%
2-Propanol	100%
Tetrahydrofuran	20%*

NOTE

Higher concentrations may only be used for limited duration applications such as column clean up at pressures < 2000 psi.

WARNING

THE ANION SELF-REGENERATING SUPPRESSOR (ASRS-ULTRA II) MUST BE OPERATED IN THE "AUTOSUPPRESSION EXTERNAL WATER MODE" WHEN USING ELUENTS CONTAINING SOLVENTS. DO NOT USE > 40% SOLVENT ON THE ASRS-ULTRA II IN THE ELECTROLYTIC MODE (POWER ON).

4.4 Making Eluents that Contain Solvents

When mixing solvents with water, remember to mix solvent with water on a volume to volume basis. If a procedure requires an eluent of 90% acetonitrile, prepare the eluent by adding 900 mL of acetonitrile to an eluent reservoir. Then add 100 mL of deionized water or eluent concentrate to the acetonitrile in the reservoir. Using this procedure to mix solvents with water will ensure that a consistent true volume/volume eluent is obtained. Premixing water with solvent will minimize the possibility of outgassing.

SAFETY

NEVER ADD THE ACETONITRILE DIRECTLY TO THE BASIC CARBONATE OR HYDROXIDE ELUENT BOTTLE. ACETONITRILE (ACN) HYDROLYZES TO AMMONIA AND ACETATE WHEN LEFT EXPOSED TO BASIC SOLUTIONS.

Add acetonitrile to basic aqueous eluents by proportioning the acetonitrile into the basic eluent with the gradient pump. This will prevent eluent contamination from acetonitrile hydrolysis. Keep the acetonitrile in a separate eluent bottle containing only acetonitrile and water.

SAFETY

IT IS POSSIBLE THAT A VOLATILE SOLVENT CAN BE “BOILED” OFF FROM THE SOLUTION; THEREFORE, WHEN PURGING OR DEGASSING ELUENTS CONTAINING SOLVENTS DO NOT PURGE OR DEGAS THE ELUENT EXCESSIVELY.

NOTE

Always degas and store all eluents in glass or plastic eluent bottles pressurized with helium. Only helium can be used to purge and degas ionic eluents containing solvents since nitrogen is soluble in solvent containing eluents.

4.5 Eluent Preparation

4.5.1 Sodium Hydroxide Eluent Concentration

a. Weight Method: When formulating eluents from 50% sodium hydroxide, Dionex recommends weighing out the required amount of 50% sodium hydroxide. Use Fisher Grade 50% sodium hydroxide. Do not use pellets.

Example: To make 1 L of 22 mM NaOH use 1.76 g of 50% sodium hydroxide: (as used in Section 5.2)

$$\text{For 22 mM:} \quad \frac{0.022 \text{ mole/L} \times 40.00 \text{ g/mole}}{50\%} = 1.76 \text{ g diluted to 1 L}$$

b. Volume Method: Although it is more difficult to make precise carbonate-free eluents for gradient analysis volumetrically; you may choose to use the following formula to determine the correct volume of 50% sodium hydroxide to be diluted.

$$g = dvr$$

Where: g = weight of sodium hydroxide required (g)
d = density of the concentrated solution (g/mL)
v = volume of the 50% sodium hydroxide required (mL)
r = % purity of the concentrated solution

Example: To make 1 L of 22 mM NaOH use 1.15 mL of 50% sodium hydroxide: (as used in Section 5.2)

$$\text{For 22 mM:} \quad \frac{0.022 \text{ mole/L} \times 40.00 \text{ g/mole}}{50\% \times 1.53 \text{ g/mL}} = 1.15 \text{ mL diluted to 1 L}$$

NOTE

This density applies to 50% NaOH. If the concentration of the NaOH solution is significantly different from 50%, the upper (weight method) calculation should be used instead.

4.5.2 Sodium Hydroxide Eluents

To make IonPac Fast Anion IIIA eluents, dilute 50% (w/w) NaOH in DI water specified in Table 4. The DI water must be degassed-deionized (DI) water having a specific resistance of 18.2 megohm-cm. Create a final volume of 1,000 mL using a volumetric flask. Avoid the introduction of carbon dioxide from the air into the aliquot of 50% NaOH and DI water.

Table 4

Dilution of 50% (w/w) NaOH to make 1000 grams of Standard IonPac Fast Anion IIIA Eluents

50% (w/w) NaOH g (mL)	Concentration of NaOH Eluent (mM)
0.40 (0.26)	5
1.76 (1.15)	22
8.00 (5.25)	100
160.00 (104.6)	2 M

4.6 Regenerant Preparation for the AMMS III

The Anion MicroMembrane Suppressor III (AMMS III) requires the use of a regenerant solution. If you are using the AMMS III instead of the Anion Self-Regenerating Suppressor ULTRA II (ASRS ULTRA II), see the "*Product Manual* for the AMMS III (Document No. 031727).

SECTION 5 - EXAMPLE APPLICATIONS

5.1 Recommendations for Optimum System Performance

The chromatograms in this section were obtained using columns that reproduced the "Production Test Chromatogram" on optimized Ion Chromatographs (see Appendix A). Different systems will differ slightly in performance due to slight differences in column sets, system void volumes, liquid sweep-out times of different components, and laboratory temperatures.

The IonPac Fast Anion IIIA is designed for the determination of inorganic anions in less than seven minutes using an isocratic hydroxide eluent delivered with an Eluent Generator. Resolution of specific analytes can be further optimized by using gradient elution. In any type of gradient elution system it is important to use eluents that produce a minimum shift in baseline conductivity during the run, as well as a fast equilibration time from one run to the next. The best choice for an eluent is sodium or potassium hydroxide as they are converted to water in the suppressor. As long as the capacity of the suppressor is not exceeded the eluent hydroxide concentration has little effect on background conductivity. For example, a gradient run could begin at 1.0 mM NaOH and end at 80 mM NaOH, with a resulting total baseline change of 1 to 2 µS.

Ensure that adequate equilibration time is allowed between runs. If downward shift in baseline is observed during the isocratic section of the chromatogram, increase the equilibration time.

You can increase the sensitivity of your system by using sample concentration techniques.

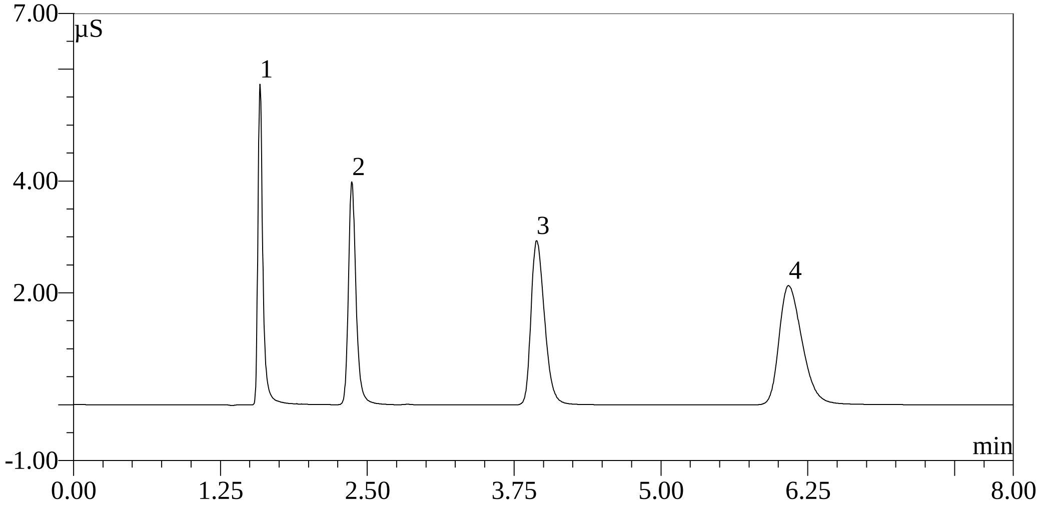
⚠ **WARNING**

Carbon dioxide readily dissolves in dilute basic solutions forming carbonate. Carbonate contamination of eluents can effect the retention times of the anions being analyzed. Eluents should be maintained under an inert helium atmosphere to avoid carbonate contamination.

5.2 Isocratic Separation with the IonPac Fast Anion IIIA

The IonPac Fast Anion IIIA provides the fast separation of fluoride, sulfate, phosphate, and citrate using an isocratic potassium hydroxide eluent, and suppressed conductivity detection. Using a 22 mM potassium hydroxide eluent delivered by an Eluent Generator, these anions can easily be separated under 7 minutes as shown in Figure 1.

Inj. Volume:	2.5 µL
Column:	(See chromatograms in Figure 1)
Eluent:	22 mM KOH
Eluent Source:	EG40 with CR-ATC
Eluent Flow Rate:	1 mL/min
Temperature:	30 °C
Detection:	Suppressed Conductivity
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS ULTRA II (2-mm)
or AAES Suppressor:	Anion Atlas Electrolytic Suppressor, AAES AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS III (2-mm)
MMS Regenerant:	50 mN H_2SO_4



	Peaks:	**ppm:**
1.	Fluoride	4
2.	Sulfate	10
3.	Phosphate	30
4.	Citrate	40

Figure 1A
Isocratic Anion Standard Separation with the IonPac Fast Anion IIIA Analytical Column

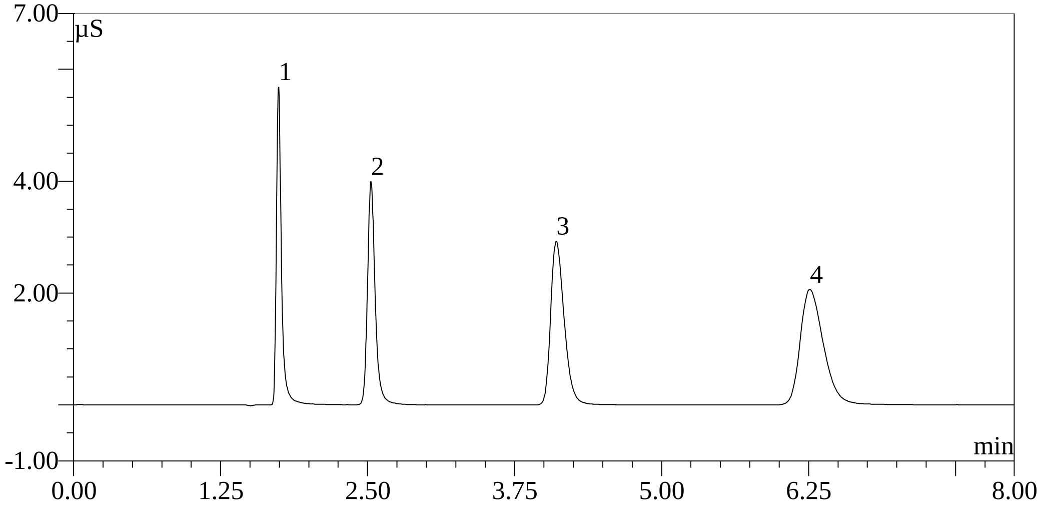
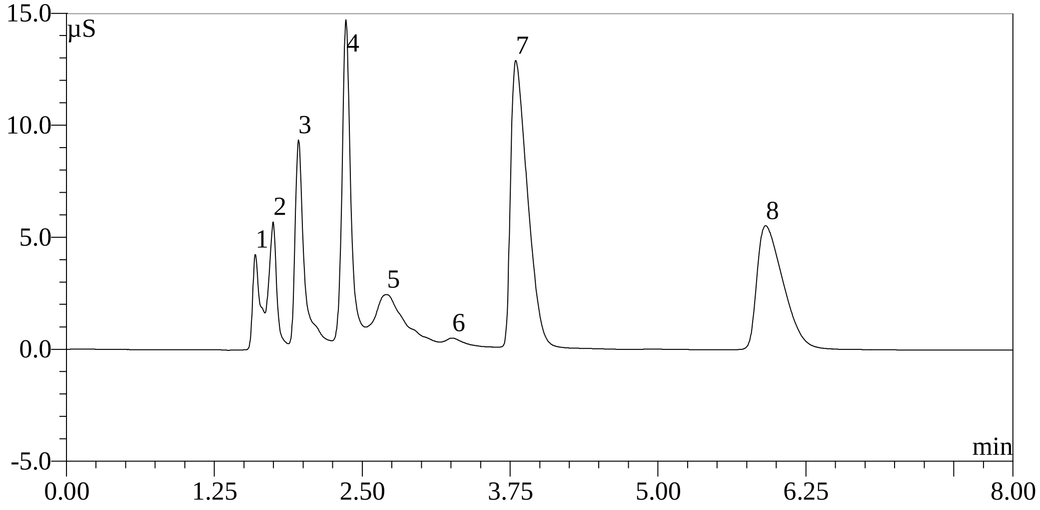


Figure 1B
Isocratic Anion Standard Separation with IonPac Fast Anion IIIA Analytical and Guard Columns

5.3 Fast Analysis of Phosphate and Citrate in a Diet Cola Sample

The IonPac Fast Anion IIIA provides the fast separation of phosphate and citrate using an isocratic potassium hydroxide eluent, and suppressed conductivity detection. Using a 22 mM potassium hydroxide eluent delivered by an Eluent Generator, these anions can easily be separated under 7 minutes in a diet cola sample, as shown in Figure 2. It is recommended that you sonicate and degas the sample to remove carbonation from the carbonated diet cola sample before injecting it into the IC (Ion Chromatograph).

Inj. Volume:	1.3 µL
Column:	(See chromatograms in Figure 2)
Eluent:	22 mM KOH
Eluent Source:	EG40 with CR-ATC
Eluent Flow Rate:	1 mL/min
Temperature:	30 °C
Detection:	Suppressed Conductivity
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS ULTRA II (2-mm)
or AAES Suppressor:	Anion Atlas Electrolytic Suppressor, AAES AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS III (2-mm)
MMS Regenerant:	50 mN H_2SO_4
Sample Preparation:	Sonicate the sample for 10 minutes



	Peaks:	ppm:
1-6.	Unknown	-
7.	Phosphate	228
8.	Citrate	175

Figure 2A
Fast Analysis of Phosphate and Citrate in a Diet Cola Sample with the IonPac Fast Anion IIIA Analytical Column

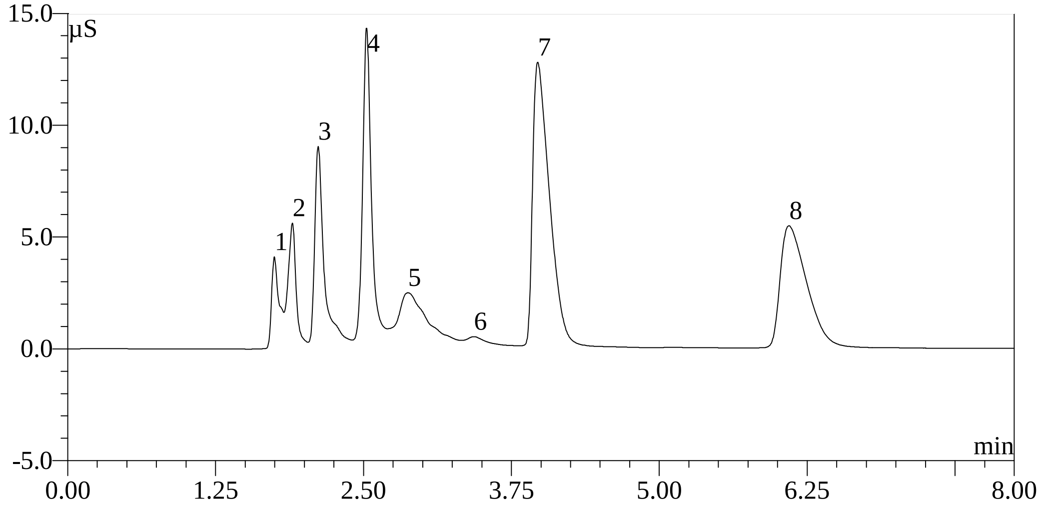
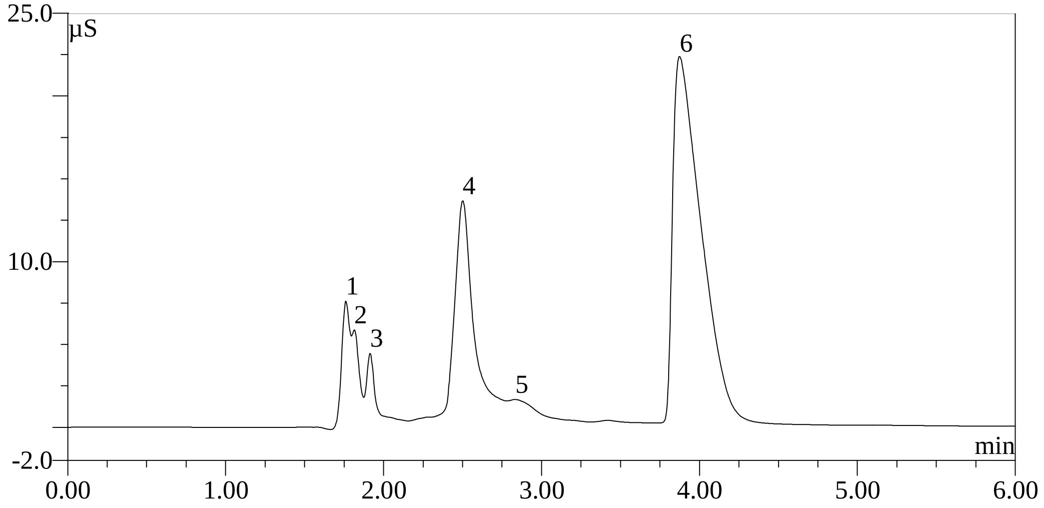


Figure 2B
Fast Analysis of Phosphate and Citrate in a Diet Cola Sample with IonPac Fast Anion IIIA Analytical and Guard Columns

5.4 Fast Analysis of Phosphate in a Cola Sample

The IonPac Fast Anion IIIA provides fast analysis of phosphate using an isocratic potassium hydroxide eluent and suppressed conductivity detection. Using a 22 mM potassium hydroxide eluent, delivered by an Eluent Generator, phosphate can easily be determined in less than 5 minutes in a cola sample; as shown in Figure 3.

Inj. Volume:	1.3 µL
Column:	IonPac Fast Anion IIIA Analytical plus Guard (3-mm)
Eluent:	22 mM KOH
Eluent Source:	EG40 with CR-ATC
Eluent Flow Rate:	1 mL/min
Temperature:	30 °C
Detection:	Suppressed Conductivity
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS ULTRA (2-mm)
or AAES Suppressor:	Anion Atlas Electrolytic Suppressor, AAES AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS III (2-mm)
MMS Regenerant:	50 mN H_2SO_4
Sample Preparation:	Sonicate the sample for 10 minutes



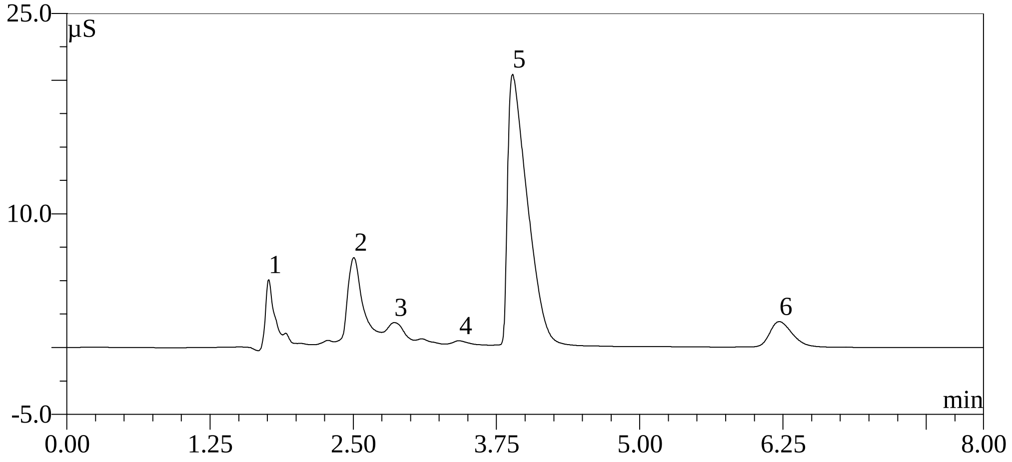
	Peaks:	ppm:
1-5	Unknown	-
6.	Phosphate	498

Figure 3
Fast Analysis of Phosphate in a Cola Sample

5.5 Fast Analysis of Phosphate and Citrate in a Cola Sample

The IonPac Fast Anion IIIA provides the fast separation of phosphate and citrate using an isocratic potassium hydroxide eluent and suppressed conductivity detection. Using a 22 mM potassium hydroxide eluent, delivered by an Eluent Generator, these anions can easily be separated under 7 minutes in a cola sample, as shown in Figure 4.

Inj. Volume:	1.3 µL
Column:	IonPac Fast Anion IIIA Analytical plus Guard (3-mm)
Eluent:	22 mM KOH
Eluent Source:	EG40 with CR-ATC
Eluent Flow Rate:	1 mL/min
Temperature:	30 °C
Detection:	Suppressed Conductivity
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS ULTRA II (2-mm)
or AAES Suppressor:	Anion Atlas Electrolytic Suppressor, AAES AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS III (2-mm)
MMS Regenerant:	50 mN H_2SO_4
Sample Preparation:	Sonicate the sample for 10 minutes



	Peaks:	ppm:
1-4.	Unknown	-
5.	Phosphate	435
6.	Citrate	53

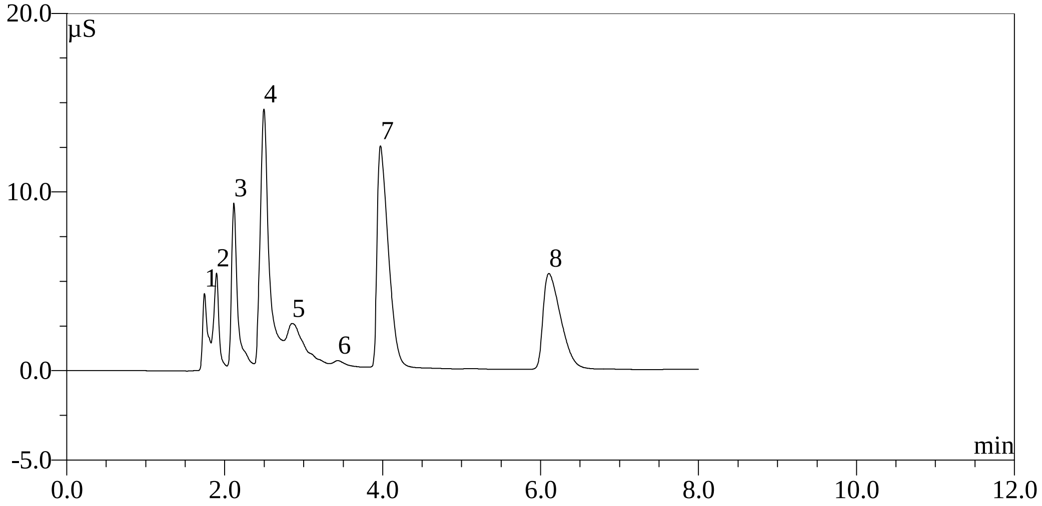
Figure 4
Fast Analysis of Phosphate and Citrate in a Cola Sample

5.6 Comparison of Isocratic and Gradient Separation of a Diet Cola Sample

The IonPac Fast Anion IIIA provides the fast separation of phosphate and citrate using an isocratic potassium hydroxide eluent and suppressed conductivity detection. Using a 22 mM potassium hydroxide eluent, delivered by an Eluent Generator, these anions can easily be separated under 7 minutes in a cola sample using Fast Anion IIIA columns, as shown in Figure 5A. A potassium hydroxide gradient can be used to provide optimum separation of the early eluting analytes as shown in Figure 5B.

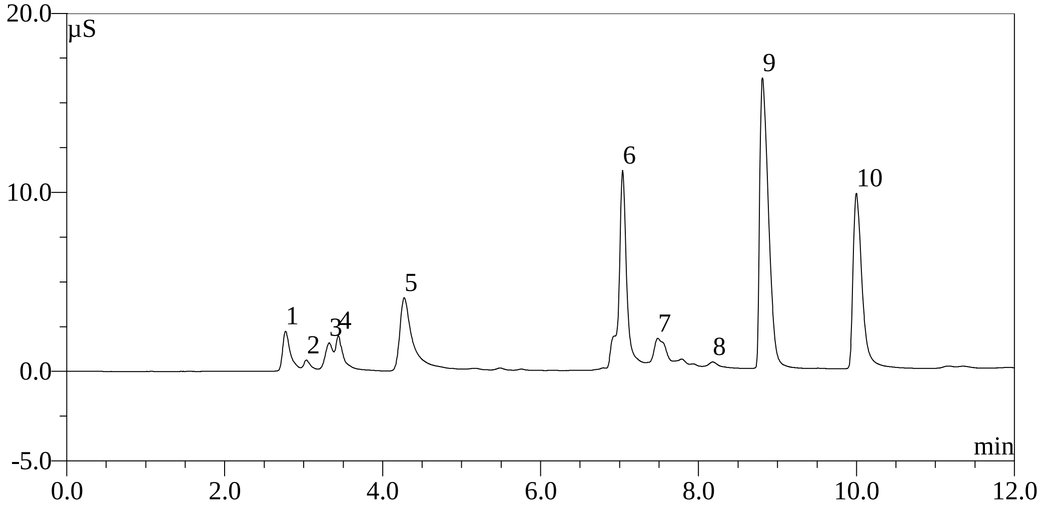
NOTE ***Under these gradient conditions, benzoate and nitrate are not resolved***.

Inj. Volume:	1.3 µL
Column:	Fast Anion IIIA Analytical plus Guard (3-mm)
Eluent:	See Chromatograms
Eluent Source:	EG40 with CR-ATC
Eluent Flow Rate:	1 mL/min
Temperature:	30 °C
Detection:	Suppressed Conductivity
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS ULTRA (2-mm)
or AAES Suppressor:	Anion Atlas Electrolytic Suppressor, AAES AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS III (2-mm)
MMS Regenerant:	50 mN H_2SO_4
Sample Preparation:	Sonicate the sample for 10 minutes



	Peaks:	ppm:
1-6.	Unknown	-
7.	Phosphate	228
8.	Citrate	175

Figure 5A - Eluent: 22 mM KOH



	Peaks:	ppm:
1-3.	Unknown	-
4.	Chloride	NQ
5.	Benzoate	167
6.	Sulfate	64
7-8.	Unknown	-
9.	Phosphate	228
10.	Citrate	175

NQ: Not Quantified

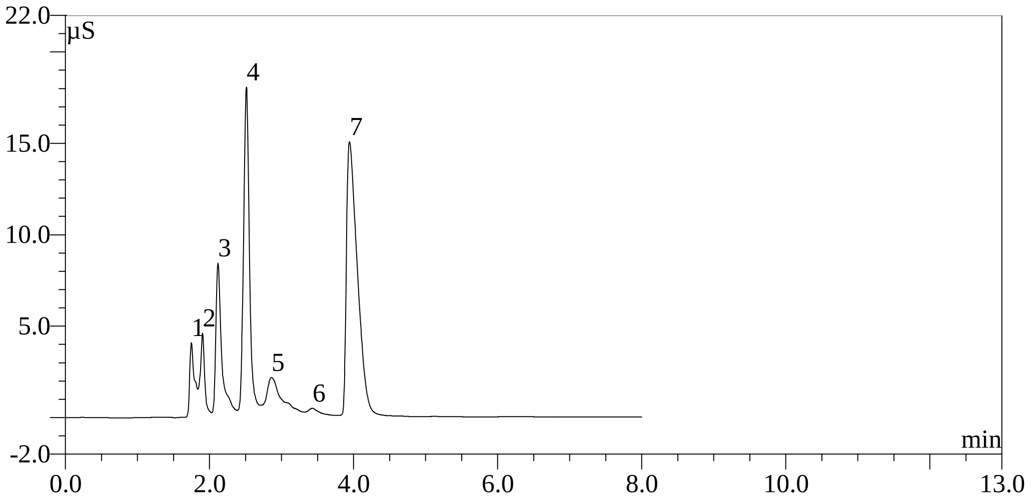
Figure 5B - Eluent: 5 mM KOH 0 to 2 min, 5 to 30 mM KOH from 2 to 8 min

5.7 Comparison of Isocratic and Gradient Separation of a Cherry Flavored Diet Cola Sample

The IonPac Fast Anion IIIA provides the fast separation of phosphate and citrate using an isocratic potassium hydroxide eluent and suppressed conductivity detection. Using a 22 mM potassium hydroxide eluent, delivered by an Eluent Generator, these anions can easily be separated under 7 minutes in a cola sample using IonPac Fast Anion IIIA columns. A potassium hydroxide gradient can be used to provide optimum separation of the early eluting anions including Benzoate and Acesulfame, found in flavored Diet Cola formulations, as shown in Figure 6B.

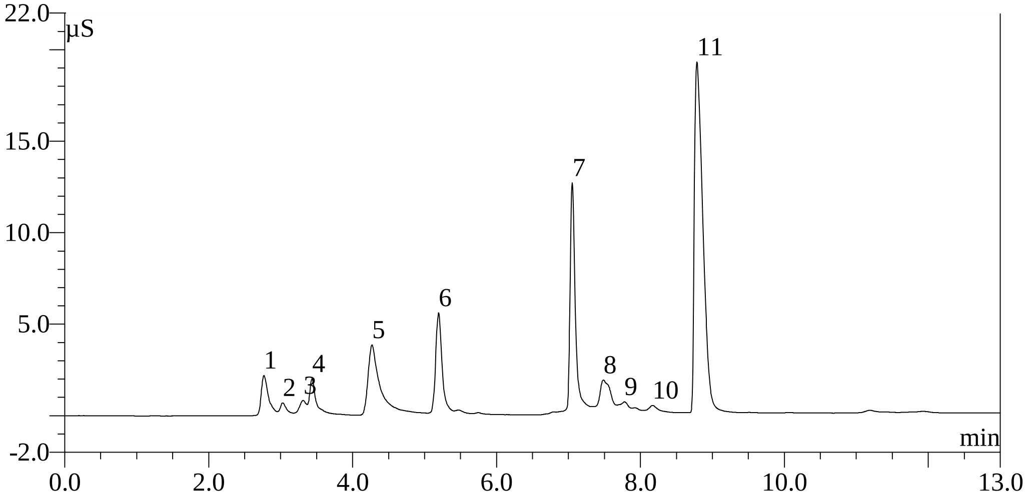
NOTE ***Under gradient conditions, acesulfame is well resolved from benzoate and sulfate.***

Inj. Volume:	1.3 µL
Column:	IonPac Fast Anion IIIA Analytical plus Guard (3-mm)
Eluent:	See chromatogram
Eluent Source:	EG40 with CR-ATC
Eluent Flow Rate:	1 mL/min
Temperature:	30 °C
Detection:	Suppressed Conductivity
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS ULTRA (2-mm)
or AAES Suppressor:	Anion Atlas Electrolytic Suppressor, AAES AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS III (2-mm)
MMS Regenerant:	50 mN H_2SO_4
Sample Preparation:	Sonicate the sample for 10 minutes



	Peaks:	ppm:
1-6	Unknown	-
7.	Phosphate	285

Figure 6A- Isocratic Separation of a Cherry Flavored Diet Cola Sample, Eluent: 22 mm KOH



	Peaks:	ppm:
1-3.	Unknown	-
4.	Chloride	NQ
5.	Benzoate	159
6.	Acesulfame	96
7.	Sulfate	52
8-10.	Unknown	-
11.	Phosphate	285

NQ: Not Quantifie-

Figure 6B- Gradient Separation of a Cherry Flavored Diet Cola Sample
Eluent: 5 mM KOH 0-2 min, 5 mM to 30 mM KOH 2-8 min

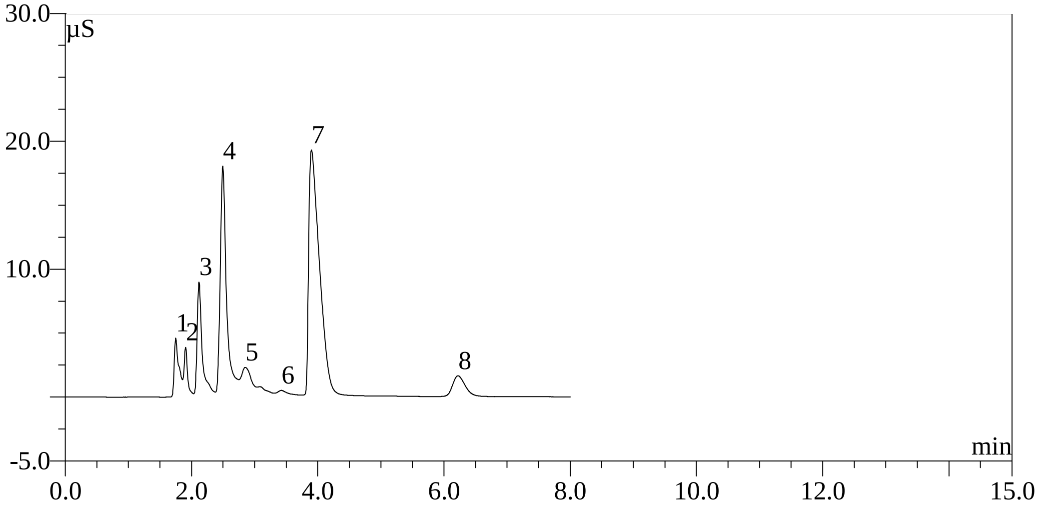
5.8 Isocratic/Gradient Separation of a Diet Cola with Splenda

The IonPac Fast Anion IIIA provides the fast separation of phosphate and citrate using an isocratic potassium hydroxide eluent and suppressed conductivity detection. Using a 22 mM potassium hydroxide eluent, delivered by an Eluent Generator, these anions can easily be separated under 7 minutes in a cola sample using IonPac Fast Anion IIIA columns. A potassium hydroxide gradient can be used to provide optimum separation of the early eluting anions including Benzoate and Acesulfame, found in Diet Cola formulations, as shown in Figure 7B.

NOTE

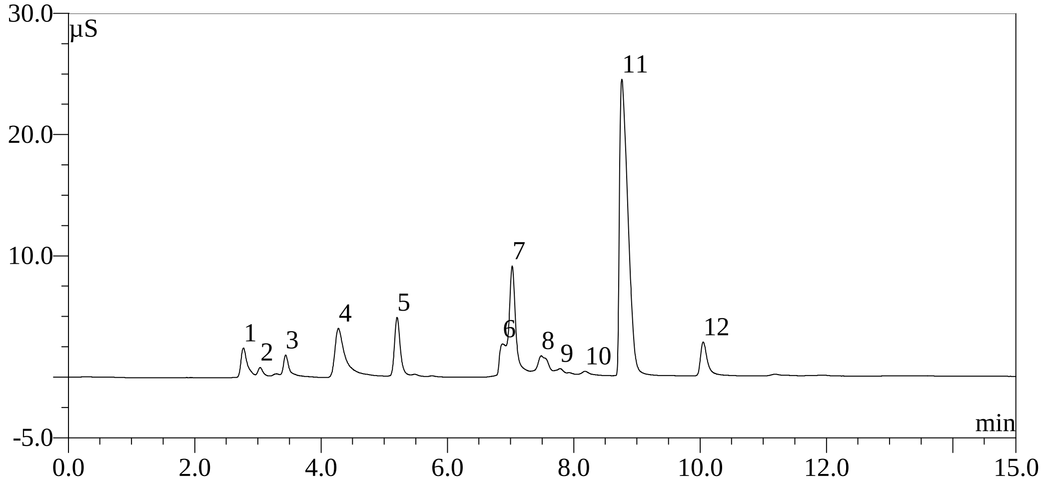
Under these conditions, acesulfame is well resolved from benzoate and sulfate.

Inj. Volume:	1.3 µL
Column:	IonPac Fast Anion IIIA Analytical plus Guard (3-mm)
Eluent:	See chromatogram
Eluent Source:	EG40 with CR-ATC
Eluent Flow Rate:	1 mL/min
Temperature:	30 °C
Detection:	Suppressed Conductivity
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS ULTRA (2-mm)
or AAES Suppressor:	Anion Atlas Electrolytic Suppressor, AAES AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS III (2-mm)
MMS Regenerant:	50 mN H_2SO_4
Sample Preparation:	Sonicate the sample for 10 minutes



	Peaks:	ppm:
1-6.	Unknown	-
7.	Phosphate	411
8.	Citrate	46

Figure 7A- Isocratic Separation of a Diet Cola with Splenda, Eluent: 22 mm KOH



	Peaks:	ppm:
1-2.	Unknown	-
3.	Chloride	7
4.	Benzoate	175
5.	Acesulfame	88
6.	Carbonate	NQ
7.	Sulfate	38
8-10.	Unknown	-
11.	Phosphate	411
12.	Citrate	46

NQ: Not Quantified

Figure 7B- Gradient Separation of a Diet Cola with Splenda
Eluent: 5 mM KOH 0-2 min, 5 mM to 30 mM KOH 2-8 min

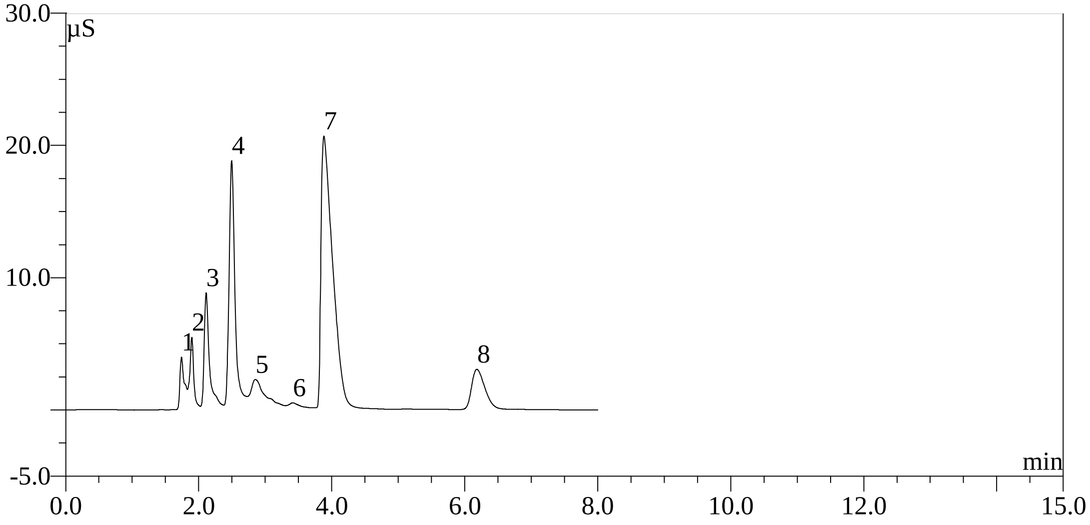
5.9 Isocratic/Gradient Separation of a Zero Cola Sample

The IonPac Fast Anion IIIA provides the fast separation of phosphate and citrate using an isocratic potassium hydroxide eluent and suppressed conductivity detection. Using a 22 mM potassium hydroxide eluent, delivered by an Eluent Generator, these anions can easily be separated under 7 minutes in a cola sample using IonPac Fast Anion IIIA columns. A potassium hydroxide gradient can be used to provide optimum separation of the early eluting anions including Benzoate and Acesulfame, found in Zero Cola formulations, as shown in Figure 8B.

NOTE

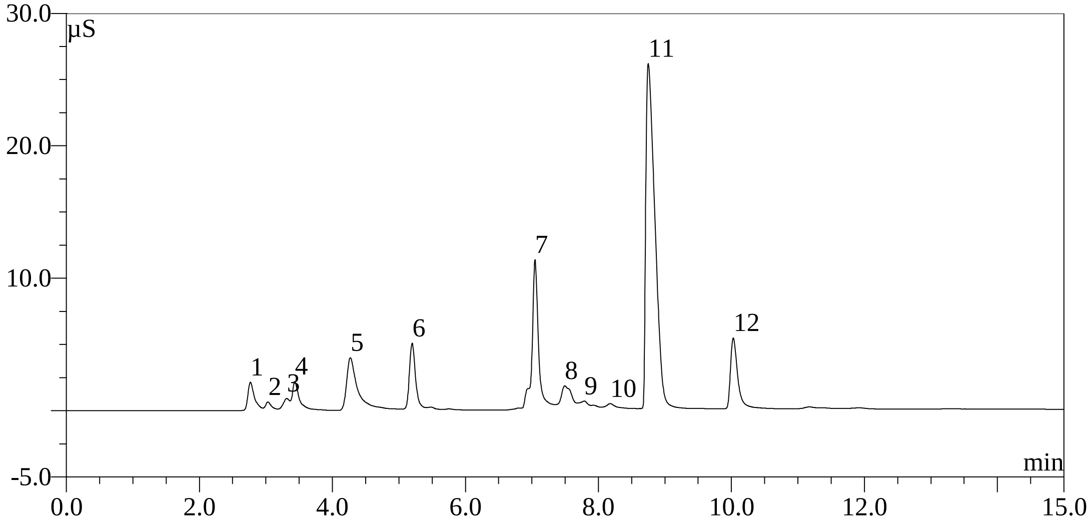
Under gradient conditions, acesulfame is well resolved from benzoate and sulfate.

Inj. Volume:	1.3 µL
Column:	IonPac Fast Anion IIIA Analytical plus Guard (3-mm)
Eluent:	See chromatogram
Eluent Source:	EG40 with CR-ATC
Eluent Flow Rate:	1 mL/min
Temperature:	30 °C
Detection:	Suppressed Conductivity
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS ULTRA (2-mm)
or AAES Suppressor:	Anion Atlas Electrolytic Suppressor, AAES AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS III (2-mm)
MMS Regenerant:	50 mN H_2SO_4
Sample Preparation:	Sonicate the sample for 10 minutes



	Peaks:	ppm:
1-6.	Unknown	-
7.	Phosphate	452
8.	Citrate	89

Figure 8A- Isocratic Separation of a Zero Cola Sample,Eluent: 22 mm KOH



	Peaks:	ppm:
1-3.	Unknown	-
4.	Chloride	NQ
5.	Benzoate	170
6.	Acesulfame	90
7.	Sulfate	62
8-10.	Unknown	-
11.	Phosphate	452
12.	Citrate	89

NQ: Not Quantified

Figure 8B- Gradient Separation of a Zero Cola Sample, Eluent: 5 mM KOH 0-2 min, 5 mM to 30 mM KOH 2-8 min

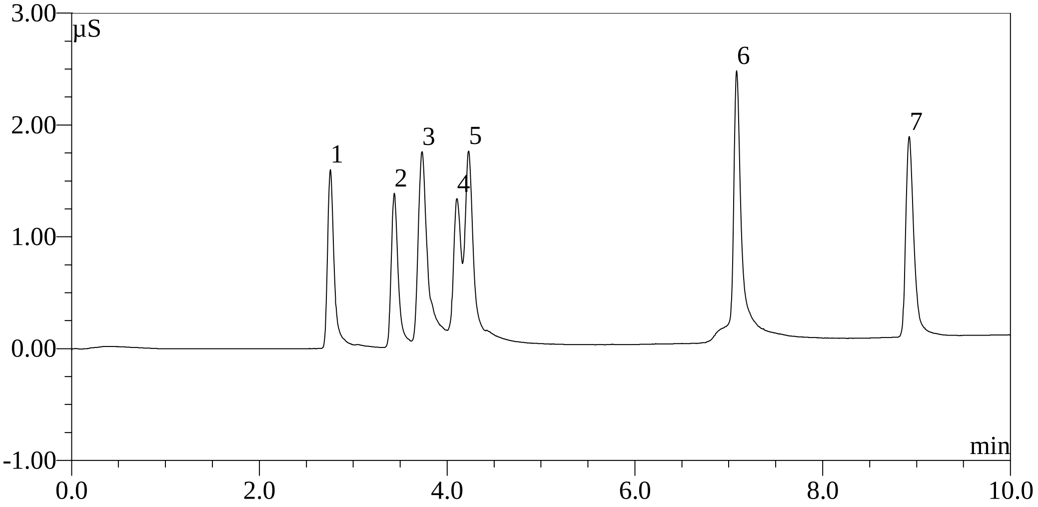
5.10 Gradient Separation of Common Inorganic Anions

Figure 9 shows the gradient separation of the common inorganic anions. These common anions can be separated in less then 10 minutes.

NOTE

The bromide and nitrite are not baseline resolved. In addition, carbonate elutes at the front of the sulfate peak.

Inj. Volume:	1.3 µL
Column:	IonPac Fast Anion IIIA Analytical plus Guard (3-mm)
Eluent:	5 mM KOH from 0 to 2 min, 5 to 30 mM from 2 to 8 min
Eluent Source:	EG40 with CR-ATC
Eluent Flow Rate:	1 mL/min
Temperature:	30 °C
Detection:	Suppressed Conductivity
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS ULTRA II (2-mm)
or AAES Suppressor:	Anion Atlas Electrolytic Suppressor, AAES AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS III (2-mm)
MMS Regenerant:	50 mN H_2SO_4



	Peaks:	**ppm:**
1.	Flouride	3
2.	Chloride	5
3.	Nitrite	10
4.	Bromide	10
5.	Nitrate	10
6.	Sulfate	10
7.	Phosphate	20

Figure 9
Gradient Separation of Common Inorganic Anions

5.11 Fast Separation of Chloride and Sulfate

Figure 10 shows the fast analysis of chloride and sulfate in less than 4 minutes. The IonPac Fast Anion IIIA Column is ideal for rapid analysis of these analytes in simple sample matrices.

Inj. Volume:	1.3 µL
Column:	IonPac Fast Anion IIIA Analytical plus Guard (3-mm)
Eluent:	15 mM KOH
Eluent Source:	EG40 with CR-ATC
Eluent Flow Rate:	1 mL/min
Temperature:	30 °C
Detection:	Suppressed Conductivity
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS ULTRA II (2-mm)
or AAES Suppressor:	Anion Atlas Electrolytic Suppressor, AAES AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS III (2-mm)
MMS Regenerant:	50 mN H_2SO_4

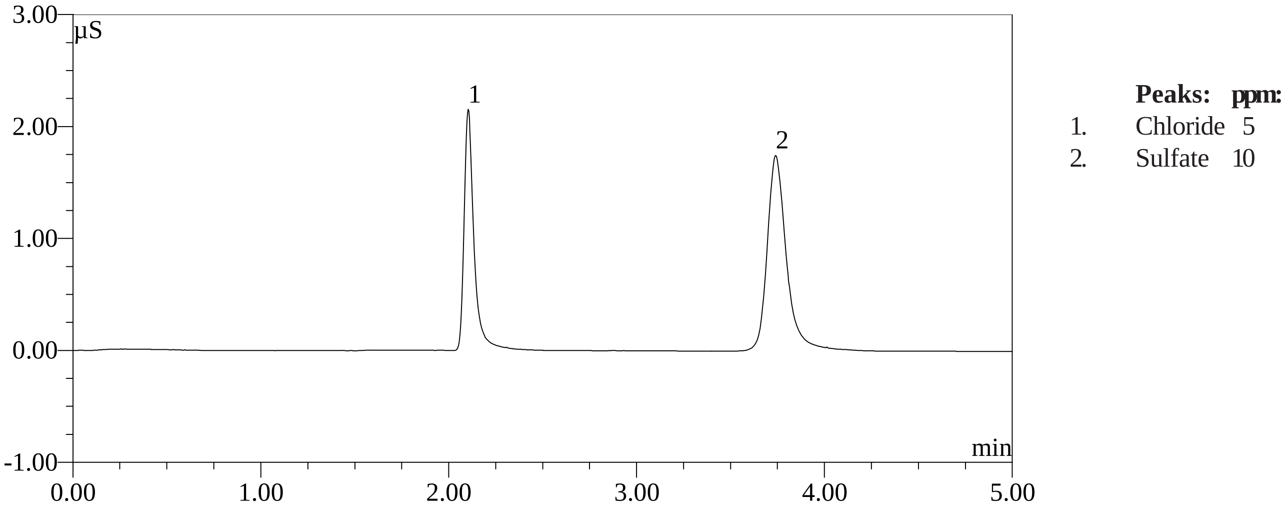


Figure 10
Fast Separation of Chloride and Sulfate

SECTION 6 - TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help solve operating problems that may arise while using IonPac Fast Anion IIIA columns. For more information on problems that originate with the Ion Chromatograph (IC) or the suppressor, refer to the Troubleshooting Guide in the appropriate product manual. For additional help contact the "Dionex North America Technical Call Center" at 1-800-DIONEX-0 (1-800-346-6390), or the nearest Dionex Office (see, "Dionex Worldwide Offices" on the Dionex Reference Library CD-ROM, P/N 053891).

Table 5
IonPac Fast Anion IIIA Troubleshooting Summary

Observation	Cause	Action
High Back Pressure	Unknown	Isolate Blocked Component
	Plugged Column Bed Supports	Replace Bed Supports Filter Eluents Filter Samples
	Other System Components	Unplug, Replace
High Background Conductivity	Contaminated Eluents	Remake Eluents
	Contaminated Trap Column	Clean Trap Column
	Contaminated AAES, ASRS or AMMS	Clean Suppressor
	Contaminated Hardware	Clean Component
Poor Resolution	Poor Efficiency Due to Large System Void Volumes	Replumb System
	Column Headspace	Replace Column
Short Retention Times	Flow Rate Too fast	Recalibrate Pump
	Conc. Incorrect Eluents	Remake Eluents
	Column Contamination	Clean Column
Poor Front End Resolution	Conc. Incorrect Eluents	Remake Eluents
	Column Overloading	Reduce Sample Size
	Sluggish Injection Valve	Service Valve
	Large System Void Volumes	Replumb System
Spurious Peaks	Sample Contaminated	Pretreat Samples
	Sluggish Injection Valve	Service Valve

6.1 High Back Pressure

6.1.1 Finding the Source of High System Pressure

Total system pressure for the IonPac Fast Anion IIIA (3-mm) Guard Column plus the IonPac Fast Anion IIIA (3-mm) Analytical Column, when using the test chromatogram conditions, should be equal or less than 2,200 psi. If the system pressure is higher than 2,200 psi, it is advisable to determine the cause of the high system pressure. The system should be operated with a High-Pressure In-Line Filter (P/N 044105) which is positioned between the Gradient Pump pressure transducer and the injection valve. Make sure you have one in place and that it is not contaminated. The maximum flow rate is 2 mL/min and the maximum pressure is 3,000 psi (20.68 MPa).

a. **Make sure that the pump is set to the correct eluent flow rate.** Higher than recommended eluent flow rates will cause higher pressure. Measure the pump flow rate if necessary with a stop watch and graduated cylinder.

b. **Determine which part of the system is causing the high pressure.** High pressure could be due to: plugged tubing or tubing with a collapsed wall, an injection valve with a clogged port, a column with particulates clogging the bed support, a clogged High-Pressure In-Line Filter, the Suppressor, or the detector cell.

To determine which part of the chromatographic system is causing the problem;

1. Disconnect the pump eluent line from the injection valve
2. Turn the pump on.
3. Watch the pressure; it should not exceed 50 psi.
4. Continue adding system components (injection valve, column(s), suppressor and detector) one by one while monitoring the system pressure.
5. The pressure should increase up to a maximum when the Guard and Analytical columns are connected (see Table 6, “Typical IonPac Fast Anion IIIA Operating Back Pressures”).

The Anion Self-Regenerating Suppressor ULTRA may add up to 100 psi (0.69 MPa). No other components should add more than 100 psi (0.69 MPa) of pressure. Refer to the appropriate manual for cleanup or replacement of the problem component.

Table 6
Typical IonPac Fast Anion IIIA Operating Back Pressures

Column	Typical Back Pressure psi (MPa)	Flow Rate mL/min
IonPac Fast Anion IIIA 3-mm Analytical	≤ 1,800 (12.41)	1.0
IonPac Fast Anion IIIA 3-mm Guard	≤ 400 (2.76)	1.0
IonPac Fast Anion IIIA 3-mm Columns	≤ 2,200 (15.17)	1.0

6.1.2 Replacing Column Bed Support Assemblies

If the column inlet bed support is determined to be the cause of the high back pressure, it should be replaced. To change the inlet bed support assembly, refer to the following instructions, and using one of the two spare inlet bed support assemblies included in the Ship Kit.

a. Disconnect the column from the system.
b. Carefully unscrew the inlet (top) column fitting. Use two open-end wrenches.
c. Remove the bed support.
d. Turn the end fitting over and tap it against a bench top or another hard--flat surface to remove the bed support and seal assembly. If the bed support must be pried out of the end fitting, use a sharp pointed object such as a pair of tweezers, but be careful that you DO NOT SCRATCH THE WALLS OF THE END FITTING.
e. Discard the old bed support assembly.
f. Make sure that the end of the column tube is clean and free of any particulate matter.
g. Place a new bed support assembly into the end fitting. seal against the bed support assembly. Use the end of the column to carefully start the bed support assembly into the end fitting.

Product	IonPac Fast Anion IIIA 3-mm Columns (P/N)
Analytical Column	062964
Guard Column	062966
Bed Support Assembly	056823
End Fitting	052809

WARNING **If the column tube end is not clean when inserted into the end fitting; particulate matter may obstruct a proper seal between the end of the column tube and the bed support assembly. If this is the case, additional tightening may not seal the column but instead damage the column tube or the end fitting.**

h. Screw the end fitting back onto the column.
i. Tighten the screw finger tight, then an additional 1/4 turn (25 in. x lb.). Tighten further only if leaks are observed.
i. Reconnect the column to the system and resume operation.

NOTE ***Replace the outlet bed support ONLY if high pressure persists after replacement of the inlet fitting.***

6.1.3 Filter Eluent

Eluents containing particulate material or bacteria may clog the column inlet bed support. Filter water, used for eluents, through a 0.45 µm filter.

6.1.4 Filter Samples

Samples containing particulate material may clog the column inlet bed support. Filter samples through a 0.45 µm filter prior to injection.

6.2 High Background or Noise

The background conductivity level for the standard eluent system is shown below.

ELUENT	EXPECTED BACKGROUND CONDUCTIVITY
22 mM NaOH	≤ 3 µS
22 mM KOH (EGC-KOH)	≤ 1.0 µS

6.2.1 Preparation of Eluents

a. Ensure the eluents and the regenerant (if used) were made correctly.
b. Ensure the eluents were made from chemicals with the recommended purity.
c. Ensure the deionized water, used to prepare the reagents, had a specific resistance of 18.2 megohm-cm.

6.2.2 A Contaminated Trap Column

High background may be caused by contamination of the ATC-HC or ATC-3 with carbonate or other anions from the eluent. Clean the ATC-HC or 4-mm ATC-3 with 100 mL of 2.0 M NaOH or 50 mL for the 2-mm ATC-3. Rinse the ATC-HC or 4-mm ATC-3 immediately with 20 mL of eluent or 10 mL of eluent for the 2-mm ATC-3 into a beaker prior to use.

6.2.3 A Contaminated CR-ATC Column

For EG50 or EG40 operation, use a CR-ATC Trap Column. Install a CR-ATC Anion Trap Column (P/N 060477) if using an Eluent Generator with EGC II KOH cartridge. If the CR-ATC becomes contaminated please refer to the "*CR-ATC Product Manual* (Document No. 031910) Section 6."

6.2.4 A Contaminated Guard or Analytical Column

Remove the IonPac Fast Anion IIIA Guard and Fast Anion IIIA Analytical Columns from the system. Install a backpressure coil that generates approx**.** 1,500 psi and continue to pump eluent. If the background conductivity decreases the column(s) is (are) the cause of the high background conductivity. To eliminate downtime, clean or replace the Fast Anion IIIA at the first sign of column performance degradation as compared to the original test chromatogram. Clean the column(s) as instructed in, “Column Cleanup” (See “Column Care”).

6.2.5 Contaminated Hardware

To eliminate hardware as the source of the high background conductivity, bypass the columns and the suppressor. Install a backpressure coil that generates approx. 1,500 psi and continue to pump eluent. Pump deionized water with a specific resistance of 18.2 megohm-cm through the system. The background conductivity should be less than 2 µS. If it is not, then check the detector/conductivity cell calibration by injecting deionized water directly into it. See the appropriate manual for details.

6.2.6 A Contaminated ASRS ULTRA II or AMMS III Suppressor

If the above items have been checked and the problem persists, the Anion Self-Regenerating Suppressor (ASRS ULTRA II) or the Anion MicroMembrane Suppressor (AMMS III) is probably causing the problem. For details on ASRS operations refer to the "*Product Manual* for ASRS ULTRA II (Document No. 031367)." For details on AMMS III operation, refer to the "*Product Manual* for AMMS III (Document No. 031727)."

a. Check the power level and alarms on the SRS Control.
b. Check the regenerant flow rate at the REGEN OUT port of the ASRS if operating in the AutoSuppression External Waster mode, the Chemical Suppression mode, or the AMMS.
c. Check the eluent flow rate.

d. If you are using an AutoRegen Accessory with the ASRS in the Chemical Suppression Mode or the AMMS, prepare fresh regenerant solution.
e. Test both the suppressor and the AutoRegen Regenerant Cartridge for contamination.

1. If the background conductivity is high after preparing fresh regenerant and bypassing the AutoRegen Regenerant Cartridge you probably need to clean or replace your ASRS or AMMS.

2. If the background conductivity is low when freshly prepared regenerant is run through the ASRS or AMMS without an AutoRegen Accessory in-line then test the AutoRegen Regenerant Cartridge to see if it is expended.

    i) Connect the freshly prepared regenerant to the AutoRegen Regenerant Cartridge.
    ii) Pump approximately 200 mL of regenerant through the AutoRegen Regenerant Cartridge to waste before recycling the regenerant back to the regenerant reservoir.

3. If the background conductivity is high after placing the AutoRegen Accessory in-line you probably need to replace the AutoRegen Regenerant Cartridge. Refer to the “*Product Manual* for AutoRegen Regenerant Cartridge Refill (Document No. 032852)."

6.2.7 A Contaminated Anion Atlas Electrolytic Suppressor (AAES)

Metal Contaminants or Precipitates

a. Turn off the power to the AAES.
b. Disconnect the analytical and guard column(s) from the injection valve and the AAES. Refer to the specific analytical column Product Manual for column cleanup procedures.
c. If you are running in the AutoSuppression External Water Mode, turn off the external water and disconnect the external water line from the AAES REGEN IN port.
d. Disconnect the liquid line from the AAES ELUENT OUT port to the cell at the cell fitting and reconnect it to the REGEN IN port.
e. Connect a temporary line from the priming block or the low-pressure tee on the isocratic or gradient pump to a container with a solution of 0.5 M oxalic acid. Pass 60 mL of this solution through the AAES using the Trap Column / Suppressor Clean-up Kit (P/N 059649) or pump this solution through the AAES at 2.0 mL/min for 30 minutes.

⚠ **CAUTION**

Bypassing internal pump manifolds when temporarily pumping high concentration cleaning solutions significantly reduces the time required to re-equilibrate the system to low concentration eluents.

f. Flush the AAES with deionized water at 2 mL/min for 30 minutes.
g. Reinstall the AAES.

Organic Contaminants

a. Turn off the power to the AAES.
b. Disconnect the analytical and guard column(s) from the injection valve and the AAES. Refer to the specific analytical column Product Manual for column cleanup procedures.
c. If you are running in the AutoSuppression External Water Mode, turn off the external water and disconnect the external water line from the AAES REGEN IN port. If you are running in the AutoSuppression Recycle Mode, proceed to D.
d. Disconnect the liquid line from the AAES ELUENT OUT port to the cell at the cell fitting and reconnect it to the REGEN IN port.
e. Connect a temporary line from the priming block or the low-pressure tee on the isocratic or gradient pump to a container with a solution of freshly prepared 10% 1.0 M H_2SO_4/90% acetonitrile. H_2SO_4/acetonitrile solutions are not stable during long term storage so this cleanup solution must be made immediately before each column cleanup. Alternatively, it can be proportioned from 1 bottle containing 1.0 M H_2SO_4 and another bottle containing 100% acetonitrile. Pass 60 mL of this solution through the AAES using the Trap Column / Suppressor Clean-up Kit (P/N 059649) or pump this solution through the AAES at 1.0 mL/min for 60 minutes.

CAUTION: Bypassing internal pump manifolds when temporarily pumping high concentration cleaning solutions significantly reduces the time required to re-equilibrate the system to low concentration eluents.

f. Flush the AAES with deionized water at 2 mL/min for 30 minutes.
g. Reinstall the AAES.

6.3 Poor Peak Resolution

Poor peak resolution can be due to any or all of the following factors.

6.3.1 Loss of Column Efficiency

a. **Peak Fronting:** Check to see if headspace has developed in the guard or analytical column. This is usually due to improper use of the column such as submitting it to high pressures. Remove the column's top end fitting (see Section 6.1.2, "Replacing Column Bed Support Assemblies"). If the resin does not fill the column body all the way to the top, it means that the resin bed has collapsed creating headspace, and the column must be replaced.

b. **Peak Tailing:** Make sure that the tubing from the injection valve to the column, from the column to the suppressor, from the suppressor to the cell, and the sample loop are connected properly.

NOTE ***In the example chromatogram, excessive peak tailing is observed due to poor plumbing of the sample loop.***

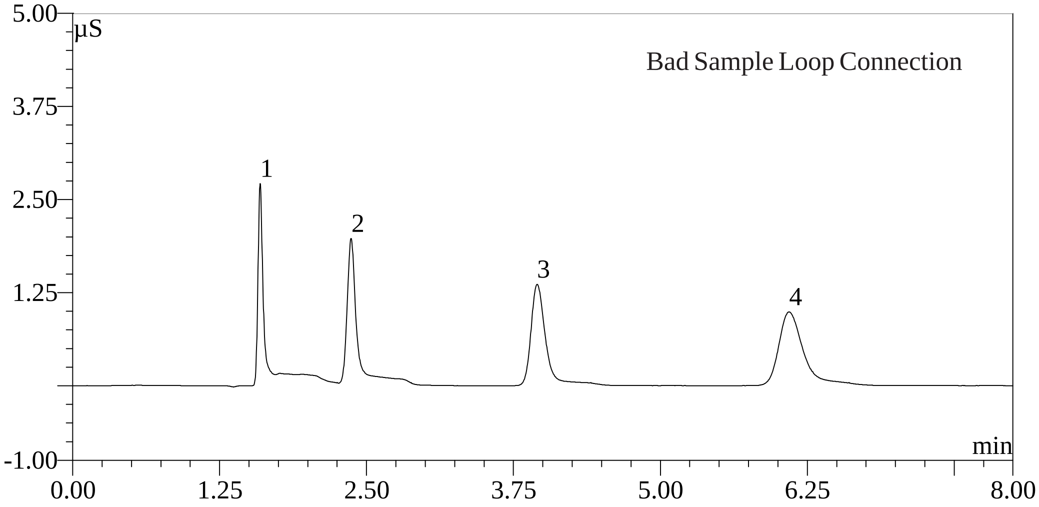


Figure 11A
Bad Sample Loop Connection

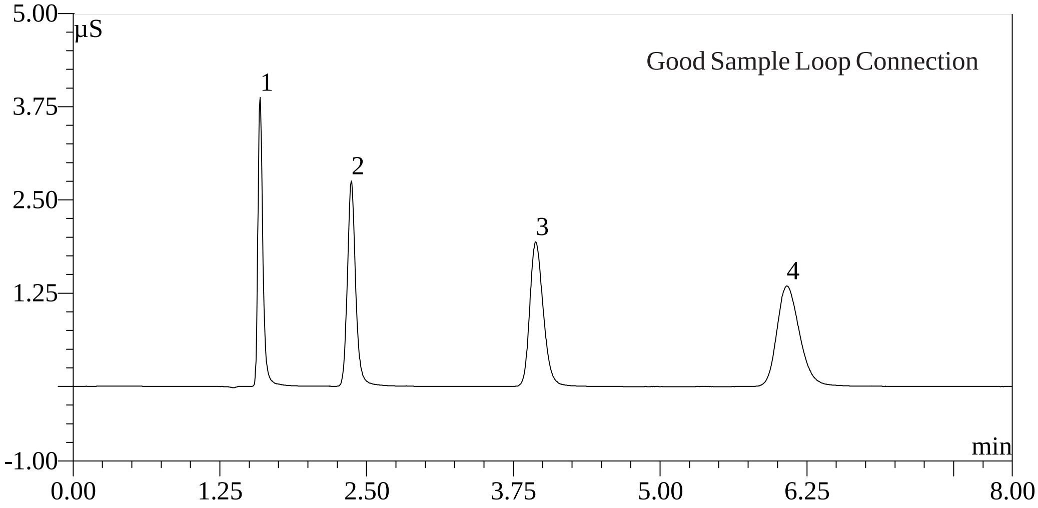


Figure 11B
Good Sample Loop Connection

c. **Symmetric Inefficient Peaks:** Extra-column effects can result in sample band dispersion making the peaks' elution less efficient. Make sure you are using PEEK tubing with an ID of no greater than 0.010" for 4-mm systems or no greater than 0.005" for 2-mm systems to make all eluent liquid line connections between the injection valve and the detector cell inlet. Cut the tubing lengths as short as possible. Check for leaks.

6.3.2 Poor Resolution Due to Shortened Retention Times

Even with adequate system and column efficiency, resolution of peaks will be compromised if analytes elute too fast.

a. **Check the flow rate:** See if the eluent flow rate is equivalent to the flow rate specified by the analytical protocol. Measure the eluent flow rate after the column using a stopwatch and graduated cylinder.

b. **Check to see if the eluent compositions and concentrations are correct:** An eluent that is too concentrated will cause the peaks to elute faster. Prepare fresh eluent. If you are using a gradient pump to proportion the eluent, components from two or three different eluent reservoirs, the resulting eluent composition may not be accurate enough for the application. Use one reservoir containing the correct

d. **Column contamination can lead to a loss of column capacity:** This is because all of the anion exchange sites will no longer be available for the sample ions. For example, polyvalent anions from the sample or metals may concentrate on the column. Refer to, "Column Cleanup" (see "Column Care"), for recommended column cleanup procedures. Possible sources of column contamination are impurities in chemicals and in the deionized water used for eluents or components of the sample matrix. Be especially careful to make sure that the recommended chemicals are used. The deionized water should have a specific resistance of 18.2 megohm-cm.

e. **Diluting the eluent will improve peak resolution, but will also increase the analytes' retention times.** If a 10% dilution of the eluent is not sufficient to obtain the desired peak resolution, or if the resulting increase in retention times is unacceptable, clean the column (see, "Column Cleanup" in "Column Care").

After cleaning the column, reinstall it in the system and let it equilibrate with eluent for about 30 minutes. No water wash is necessary. The column is equilibrated when consecutive injections of the standard give reproducible retention times. The original column capacity should be restored by this treatment sense the contaminants should be eluted from the column. If you need assistance in solving resolution problems contact the "Dionex North America Technical Call Center" at 1-800-DIONEX-0 (1-800-346-6390), or the nearest Dionex Office (see "Dionex Worldwide Offices").

6.3.3 Loss of Front End Resolution

If poor resolution or efficiency is observed for the peaks eluting near the system void volume compared to the later eluting peaks then check for the following possible problems:

a. **Improper eluent concentration:** Remake the eluent as required for your application. Ensure that the water and chemicals used are of the required purity.

b. **Column overloading:** Reduce the amount of sample ions being injected onto the analytical column by either diluting the sample or injecting a smaller volume onto the column.

c. **Sluggish operation of the injection valve:** Check the air pressure and make sure there are no gas leaks or partially plugged port faces. Refer to the valve manual for instructions.

d. **Improperly swept out volumes anywhere in the system prior to the guard and analytical columns:** Swap components, one at a time, in the system prior to the analytical column and test for front-end resolution after every system change. Use the shortest tubing lengths possible.

6.3.4 Spurious Peaks

a. **The columns may be contaminated:** If the samples contain an appreciable level of polyvalent ions and the column is used with a weak eluent system the retention times for the analytes will then decrease and be spurious causing inefficient (broad) peaks that can show up at unexpected times. Clean the column as indicated in "Column Cleanup" (see "Column Care").

If you need assistance in determining the best way to clean strongly retained solutes in your specific sample matrix from the IonPac Fast Anion IIIA columns contact the "North America Technical Call Center" at 1-800-DIONEX-0 (1-800-346-6390), or the nearest Dionex Office (see "Dionex Worldwide Offices" on the Dionex Reference Library CD-ROM, P/N 053891).

b. **The injection valve may need maintenance:** When an injection valve is actuated the possibility of creating a baseline disturbance exists. This baseline upset can show up as a peak of varying size and shape. This will occur when the injection valve needs to be cleaned or retorqued (see valve manual). Check to see that there are no restrictions in the tubing connected to the valve. In addition, check the valve port faces for blockage and replace them if necessary. Refer to the Valve Manual for troubleshooting and service procedures. Small baseline disturbances at the beginning or at the end of the chromatogram can be overlooked as long as they do not interfere with the quantification of the peaks of interest.

APPENDIX A - COLUMN CARE

A.1 Recommended Operation Pressures

Operating a column above its recommended pressure limit can cause irreversible loss of column performance. The maximum recommended operating pressure for IonPac Fast Anion IIIA column is 3,000 psi (20.68 MPa).

A.2 Column Start-Up

The column is shipped with 10 mM sodium bicarbonate as the storage solution.

Prepare the eluent shown on the test chromatogram, install the column in the chromatography module and test the column performance under the conditions described in the test chromatogram. Continue making injections of the test standard until consecutive injections of the standard give reproducible retention times. Equilibration is complete when consecutive injections of the standard give reproducible retention times.

A.3 Column Storage

For short-term storage (< 1 week), use Eluent, for long-term storage (> 1 week), use 100 mM Sodium Borate for the column storage solution. Flush the column for a minimum of 10 minutes with the storage solution. Cap both ends securely, using the plugs supplied with the column.

A.4 Column Cleanup

The following column cleanup protocols have been divided into three general isocratic protocols to remove acid-soluble, base-soluble or organic contaminants. They can be combined into one gradient protocol if desired but the following precautions should be observed.

Always ensure that the cleanup protocol used does not switch between eluents which may create high pressure eluent interface zones in the column. High pressure zones can disrupt the uniformity of the packing of the column bed and irreversibly damage the performance of the column. High pressure zones in the column can be created by pumping successive eluents through the column that are not miscible, that have eluent components in one eluent that will precipitate out in the other eluent or by using an acid eluent followed by a base eluent which may create a neutralization pressure band. The precipitation of the salts in solvents during column rinses can result in very high pressure zones. High viscosity mixing zones can be created between two eluents having solvents with a very high energy of mixing.

When in doubt, always include short column rinse steps to reduce the solvent content of the eluent to $\leq$ 5% levels and the ionic strength of the eluent to $\leq$ 50 mM levels to avoid creating high pressure zones in the column that may disrupt the uniformity of the column packing.

A.4.1 Choosing the Appropriate Cleanup Solution

a. Concentrated hydroxide solutions such as a 10X concentrate of the most concentrated eluent used in the application is sufficient to remove hydrophilic contamination of low valence.

b. Concentrated acid solutions such as 1 to 3 M HCl, remove high valence hydrophilic ions by ion suppression and elution by the chloride ion.

c. Metal contamination often results in asymmetric peak shapes and/or variable analyte recoveries. For example, iron or aluminum contamination often results in tailing of sulfate and phosphate. Aluminum contamination can also result in low phosphate recoveries.

Concentrated acid solutions such as 1 to 3 M HCl remove a variety of metals. If after acid treatment, the chromatography still suggests metal contamination, treatment with chelating acids such as 0.2 M oxalic acid is recommended.

d. Organic solvents can be used alone if the contamination is nonionic and hydrophobic. The degree of nonpolar character of the solvent should be increased as the degree of hydrophobicity of the contamination within the range of acceptable solvents listed in Table 3, HPLC Solvents for Use with IonPac Fast Anion IIIA Column.

e. Concentrated acid solutions such as 1 to 3 M HCl can be used with compatible organic solvents to remove contamination that is ionic and hydrophobic. The acid suppresses ionization and ion exchange interactions of the contamination with the resin. The organic solvent then removes the subsequent nonionic and hydrophobic contamination. See Section D above.

A frequently used cleanup solution is 200 mM HCl in 80% acetonitrile. This solution must be made immediately before use because the acetonitrile will decompose in the acid solution during long term storage.

f. Regardless of the cleanup solution chosen, use the following cleanup procedure in, “Column Cleanup Procedure”, to clean the Fast Anion IIIA Analytical and Guard Columns.

A.4.2 Column Cleanup Procedure

a. Prepare a 500 mL solution of the appropriate cleanup solution using the guidelines in, “Choosing the Appropriate Cleanup Solution”.

b. Disconnect the ASRS ULTRA II, AAES, or AMMS III from the IonPac Fast Anion IIIA Analytical Column. If your system is configured with both a guard column and an analytical column, reverse the order of the guard and analytical column in the eluent flow path. Double check that the eluent flows in the direction designated on each of the column labels.

WARNING

When cleaning an analytical column and a guard column in series, ensure that the guard column is placed after the analytical column in the eluent flow path. Contaminants that have accumulated on the guard column can be eluted onto the analytical column and irreversibly damage it. If in doubt, clean each column separately.

c. Set the pump flow rate to 1.0 mL/min for an IonPac Fast Anion IIIA Analytical or Guard Column.

d. Rinse the column for 10 minutes with deionized water before pumping the chosen cleanup solution over the column.

e. Pump the cleanup solution through the column for at least 60 minutes.

f. Rinse the column for 10 minutes with deionized water before pumping eluent over the column.

g. Equilibrate the column(s) with eluent for at least 60 minutes before resuming normal operation.

h. Reconnect the ASRS ULTRA II, AAES, or AMMS III to the Fast Anion IIIA Analytical Column and place the guard column in line between the injection valve and the analytical column if your system was originally configured with a guard column.

APPENDIX B - ION CHROMATOGRAPHY SYSTEM REQUIREMENTS

As shown below, the proper configuration of the 3-mm system injection volume, mass loading, void volume, and flow rate is based on the ratio of the 3-mm to 4-mm column cross-sectional area, which is a factor of 0.50.

Table B1 - Configuration

CONFIGURATION	3-mm
Eluent Flow Rate	1.0 mL/min
SRS Suppressor	ASRS ULTRA II (2 mm) (P/N 061562)
AES Suppressor	AAES (P/N 056116)
MMS Suppressor	AMMS III (2 mm) (P/N 056751)
NOTE: ***Do not operate suppressors over 40 °C. If application requires a higher temperature, place suppressor outside of chromatographic oven.***	
Injection Loop	1 - 25 µL: Use the Rheodyne Microinjection Valve, Model No. 9126 DIONEX P/N 044697) for full loop injections <15 µL.
System Void Volume	Eliminate switching valves, couplers and the GM 3 Gradient Mixer. Use only the 2 mm GM-4 Mixer (P/N 049135).
Pumps	Use the GS50/GP50/GP40/IP20/IP25 in Microbore Configuration with a Microbore GM-4 (2 mm) Gradient Mixer. The GPM 2 can be used for 2 mm isocratic chromatography at flow rates of 0.5 mL/min or greater but cannot be used for 2 mm gradient chromatography ***NOTE:*** ***The use of an EG40 (P/N 053920) or EG50 (P/N 060585) with an EGC II KOH cartridge (P/N 058900) for gradient applications is highly recommended for minimum baseline change when performing eluent step changes or gradients.***
Detectors	CD20, CD25, CD25A, ED40, ED50, or ED50A Conductivity Cell with DS3 P/N 044130 or Conductivity Cell with shield P/N 044132 CDM 2, CDM 3 Cell P/N 042770 Replace the TS 1 with the TS 2 (P/N 043117) on the CDM 2 or the CDM 3. The TS-2 has been optimized for 2 mm operation. Do not use the TS 2 or the TS 1 with the ED40/ED50/ED50A or the CD20/CD25/CD25A.

Table B2
Tubing Back Pressures

Color	Dionex P/N	ID Inches	ID cm	Volume µL/cm	Back Presure psi/ft at 1 mL/min	Back Presure psi/ft at 0.25 mL/min	Back Presure psi/cm at 1 mL/min
Green	044777	0.030	0.076	4.560	0.086	0.021	0.003
Orange	042855	0.020	0.051	2.027	0.435	0.109	0.015
Blue	049714	0.013	0.033	0.856	2.437	0.609	0.081
Black	042690	0.010	0.025	0.507	6.960	1.740	0.232
Red	044221	0.005	0.013	0.127	111.360	27.840	3.712
Yellow	049715	0.003	0.008	0.046	859.259	214.815	28.642